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ENVIRONMENTAL DURABILITY TESTING OF STRUCTURAL ADHESIVES PART II, BR-127/FM-123

D. Robert Askins

University of Dayton Research Institute
Dayton, Ohio 45469

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Weldon Scardino

MR. WELDON SCARDINO
Specialty Materials
Materials Engineering Branch

Albert Olevitch

ALBERT OLEVITCH, Chief
Non-Metals Engineering Branch
Systems Support Division

Martin A. Alewine

MARTIN A. ALEWINE, JR., COL.
Acting Chief, Systems Support Division

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A program has been conducted to investigate the durability of a 250°F (121°C) curing modified epoxy structural adhesive in an elevated temperature, high humidity environment while under stress.

The results indicate that, for shear stress levels low enough to preclude fracture of the adherend surface oxide layer [below 2000 psi (13.9 MPa)], the FM-123-2/BR-127 adhesive/primer system provides long-term durability at 120°F (49°C) and 95-100% R.H. on

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both phosphoric acid anodized (PAA) and optimized Forest Products Laboratory (OFPL) etched surfaces. The change in slope of the environmental stress-rupture time-to-failure curve for the PAA specimens seems to correlate well with the stress level at which oxide fracture would be expected to occur [about 3000 psi (20.65 MPa)] and may indicate a different degradation mechanism above this stress level than below. The effect of temperature upon the durability of the adhesive joints tested in this investigation is to reduce durability with increasing temperature, as was to be expected. At higher aging temperatures, the interface seems to be more susceptible to degradation than the adhesive, while at lower temperatures the reverse is true.

This is the second part of a continuing program to evaluate the stressed durability of adhesive/primer systems of immediate interest to the Air Force.

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PREFACE

This report covers work performed during the period from May 1976 to May 1978 under Air Force Contracts F33615-76-C-5034 and F33615-78-C-5002, Project Number 7381. This is the second report to be published in a series dealing with the same general theme. The first report describes the environmental durability testing of AF-143/EC-3917 and PL-729-3/PL-278, 350°F curing adhesive systems. The work was administered under the direction of the Systems Support Division of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. Mr. Weldon Scardino (AFML/MXE) acted as Project Engineer.

The Principal Investigator on this investigation was William E. Berner. The major portion of the laboratory work was conducted by John Dues, research technician.

This report was submitted by the author in December 1978. The contractor's report number is UDR-TR-78-53.

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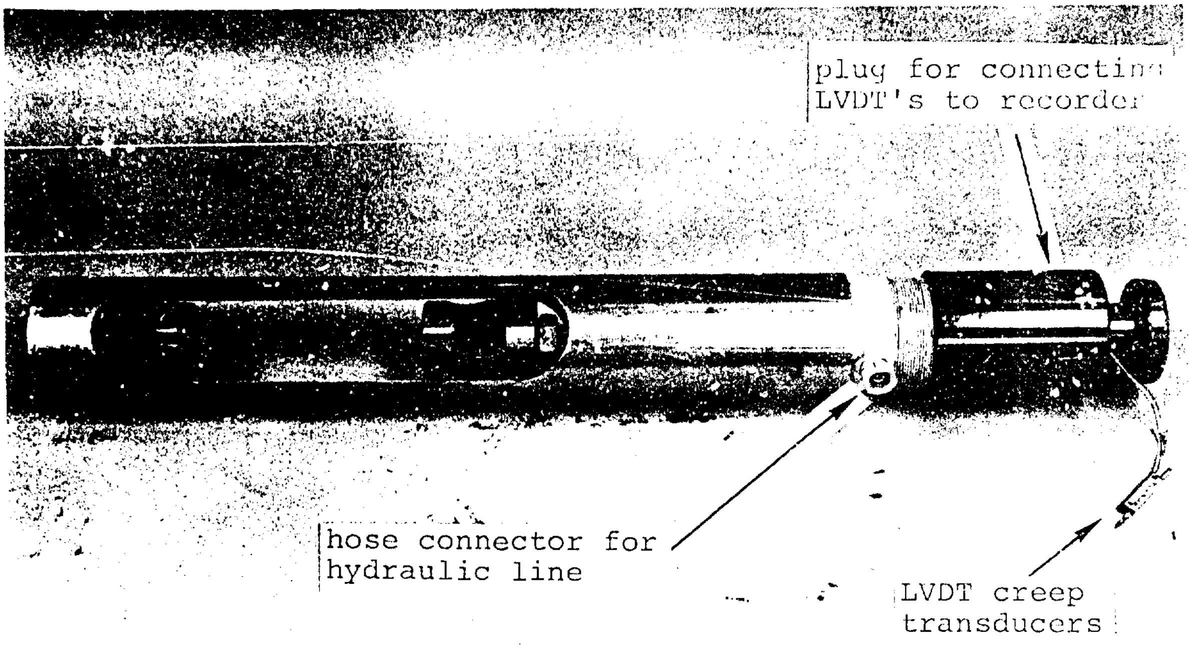
SECTION I
INTRODUCTION

The University of Dayton Research Institute has been conducting investigations into the durability of adhesives and adhesive bonded structures for several years. Much of this work in recent years has involved the use of environmental stress-rupture testing of lap shear joints. The test apparatus which permits the measurement of the durability of bonded joints while exposed to elevated temperature and humidity under a controlled stress level was designed and constructed by the University of Dayton and has been in service for several years. It permits time-to-failure measurements on stressed adhesive bonds in adverse environments and also has the capability of measuring joint deformation as a function of exposure time. AFML-TR-78-35, Part I, described an investigation of the durability of two 350°F (177°C) curing adhesive systems on acid etched and anodized adherend surfaces (PL729 and AF143 adhesives with 2024T3 bare and 7075T6 bare adherends).

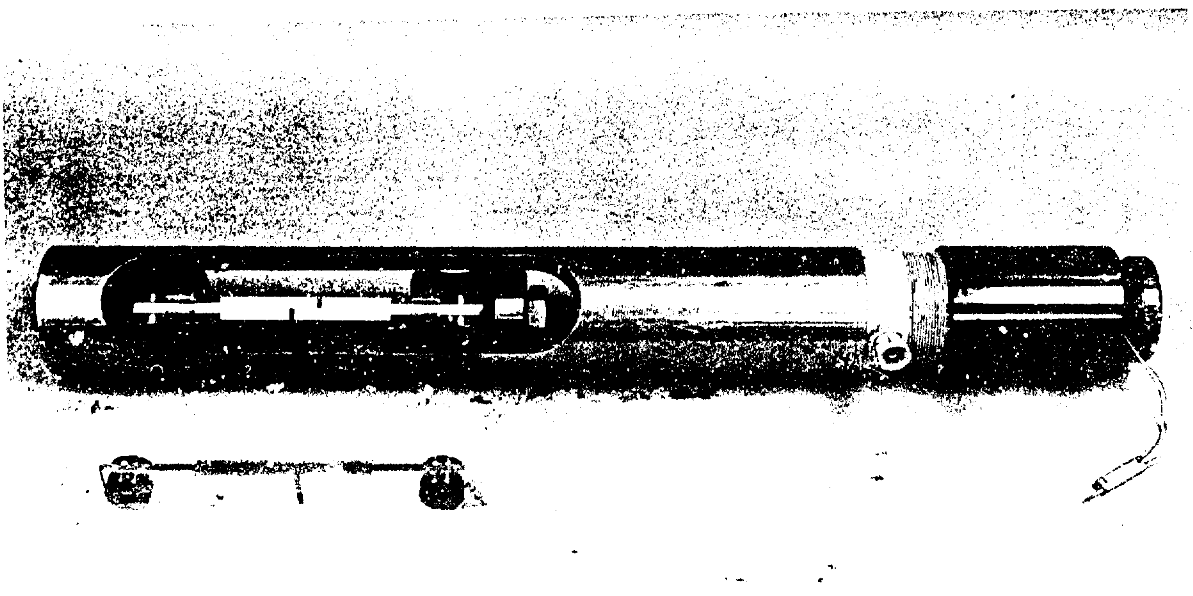
In the investigation reported here, a 250°F (121°C) curing adhesive system has been investigated on both acid etched and phosphoric acid anodized adherend surfaces. Durability tests using the environmental stress-rupture apparatus have been conducted at two different temperatures to compare the effect of exposure temperature on the performance of lap shear joints.

SECTION II
DURABILITY TEST APPARATUS

The durability test apparatus provides the capability of conducting environmental exposures on specimens subjected to a constant tensile load during the exposure period. The environment can be controlled between 95°F (35°C) and 200°F (93°C) and between 40% and 95% R.H. Loads are applied hydraulically and can be controlled to within ± 5 lbs (± 22 N) over a range from 0 to 2500 lbs (0 to 11,125 N). Figures 1 to 3 illustrate the test apparatus and specimen mounting cells. An adhesive lap shear specimen of the type used in this program is shown mounted and also lying beside the test cell in Figure 1b. The tester can accommodate 12 specimens simultaneously. Although all 12 are exposed to the same temperature and humidity conditions, the load on each can be independently controlled. The exposure cabinet is a standard Blue M humidity cabinet, model AC-7502HA-1, which has 12 holes cut through the door for insertion of the test cells. Each test cell permits free access of the environment to the test specimen. Small Linear-Variable-Differential-Transformers (LVDT) transducers are mounted in the hydraulic loading heads of each cell. These transducers permit continuous recording of specimen creep deformation during exposure. The creep measurement capability was not utilized in this investigation, however; only time-to-rupture was recorded.



(a) Empty cell



(b) Cell with mounted specimen

Figure 1. Specimen Mounting Cells For the Durability Test Apparatus

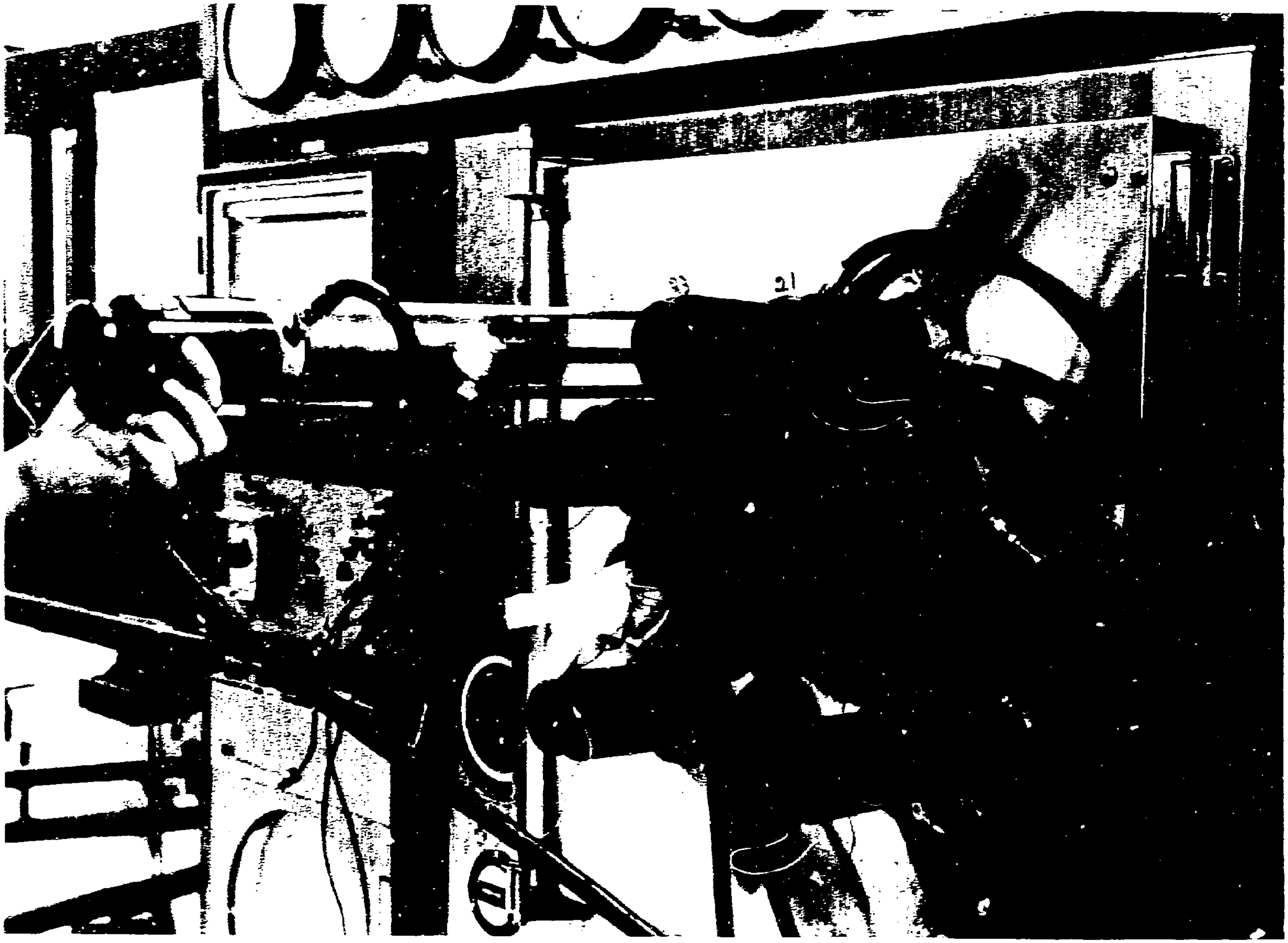


Figure 2. Specimen Mounting Cell Being Inserted Into Humidity Cabinet

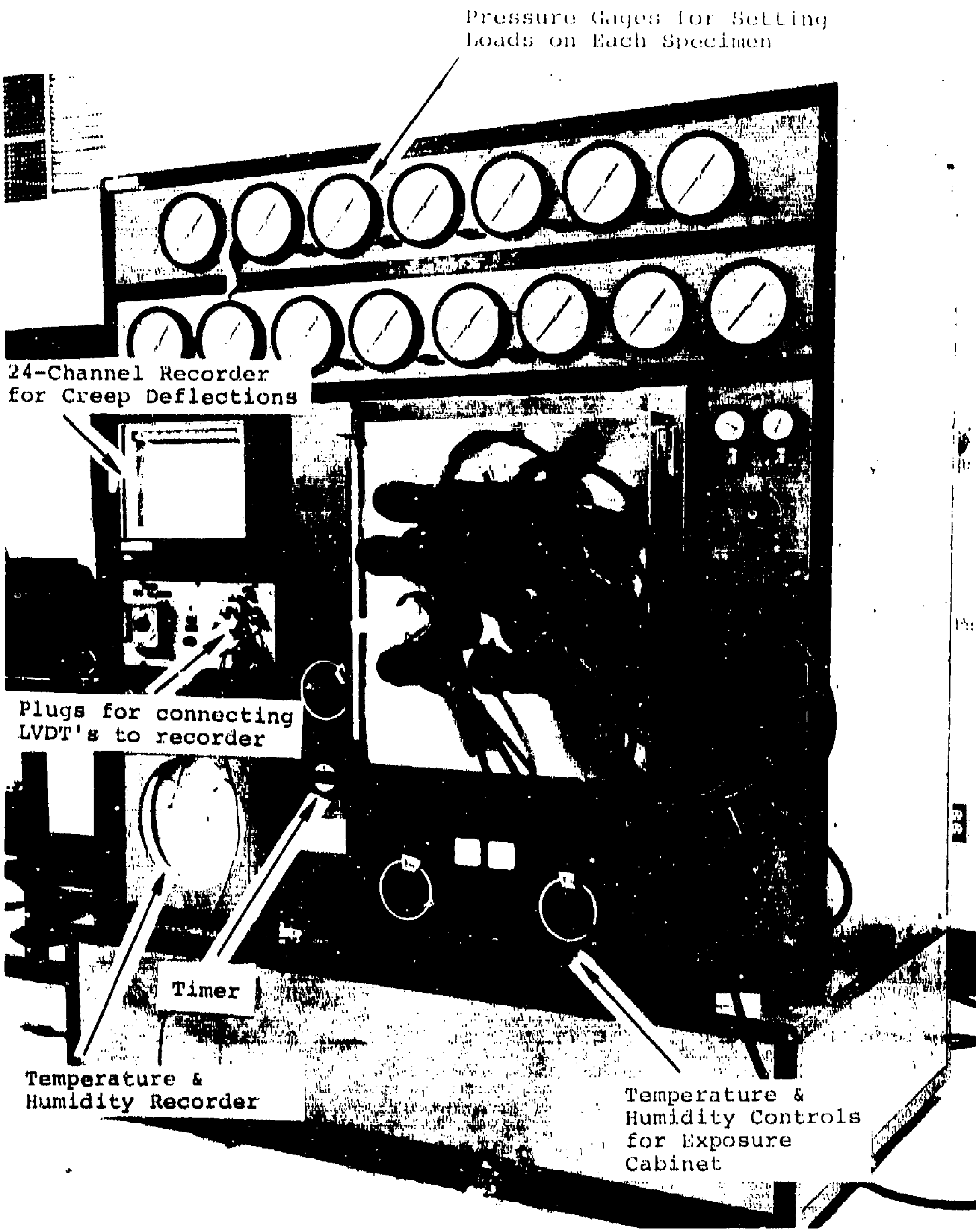


Figure 3. Overall View of Durability Test Apparatus

SECTION III
EXPERIMENTAL PROGRAM

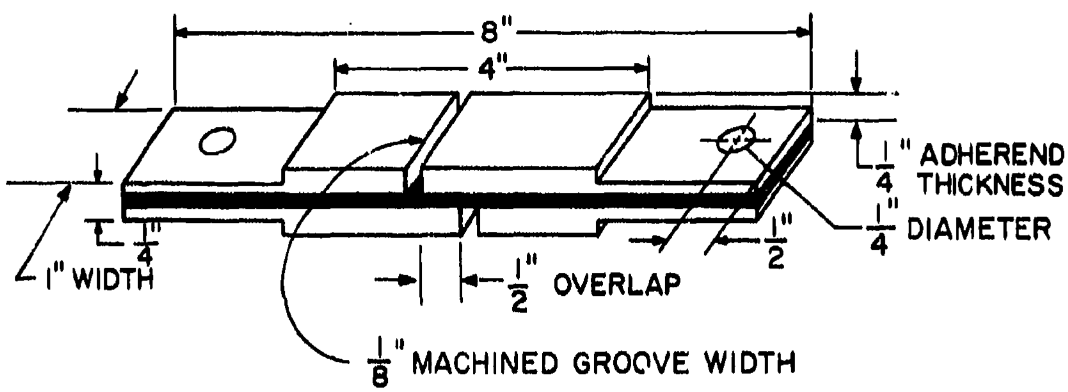
1. MATERIALS

A 250°F (121°C) curing modified epoxy structural adhesive (FM123-2 by American Cyanimid) has been evaluated. The adhesive is nominally 0.060 lb/ft^2 (0.29 Kg/m^2) and is nominally 0.011 inch (0.028 cm) thick. It is supported on a dacron mat carrier. This adhesive was used in combination with a corrosion-inhibiting primer (BR 127 by American Cyanimid). Due to the length of time over which this investigation was conducted, three different batches of adhesive were ordered and used. The first batch was used to prepare the specimens on an optimized FPL etched adherend. These were subsequently tested for durability in the environmental stress-rupture chamber at 140°F (60°C). The second batch of adhesive was used to prepare the specimens on a phosphoric acid anodized adherend. These were tested for environmental stress-durability at 120°F (49°C). The third batch of adhesive was used to prepare specimens with an optimized FPL etched adherend and were tested for environmental durability at 120°F (49°C).

All of the specimens in this investigation were prepared with 7075T6 bare aluminum adherends. These adherends were 0.250 inch (0.64 cm) thick and were used to prepare machined single lap shear specimens (also known as blister shear specimens). Figure 4 illustrates this specimen.

Two surface preparations were used during the course of this work; an optimized FPL etch and a phosphoric acid anodized surface preparation. The former is the same as that described in Boeing process specification BAC 5514, while the latter is the same as that described in Boeing process specification BAC 5555 and ARP*1564. The central features of these processes are presented in Appendix B.

*Aerospace Recommended Practice (SAE)



0.250 inch (0.64cm), thick adherend, machined single lap shear specimen

Figure 4. Single Lap Shear Adhesive Specimen

2. SPECIMEN FABRICATION

Specimen fabrication procedures can be separated into three general phases. The first phase deals with adherend surface preparation, the second with the panel bonding operation, and the third with the machining of the bonded panel into individual test specimens. Details of each of these three phases are presented respectively in Appendices B, C, and D.

3. TEST PLAN

Three types of tests were conducted on the lap shear specimens in this investigation. The first was a simple static test on the as-fabricated, dry specimens at three different temperatures; 72°F (22°C), 120°F (49°C) or 140°F (60°C), and 250°F (121°C). The second type was also a simple static test at 72°F (22°C) on specimens which had been exposed to elevated temperature, high-humidity aging [120°F/49°C or 140°F/60°C and 100% R.H.] for 28 and 100 days prior to testing. The third type of test was an environmental stress-rupture test in which the lap shear specimens were loaded to various stress levels and exposed to elevated temperature, 95-100% R.H. environment until failure. The elevated temperature during these environmental stress-rupture tests was either 120°F (49°C) or 140°F (60°C). If no failure had occurred within 2400 hours, the specimens were removed from the environmental durability tester and tested statically at 72°F (22°C) for residual strength. The stresses imposed during the environmental durability exposures varied between 20% and 80% of the ultimate strength obtained in the dry static tests at either 120°F (49°C) or 140°F (60°C). All of the lap shear tests conducted on specimens which had been humidity aged (either the static or residual strength tests) were completed within 30 minutes after the specimen was removed from the environmental chamber. Additionally, each of these specimens was wrapped with a wet cloth to prevent dryout during this period.

SECTION IV
DISCUSSION OF RESULTS

Tables 1-6 present the test results obtained during this investigation. Tables 1, 3, and 5 present the average ultimate strength values obtained from the static lap shear tests. Tables 2, 4, and 6 present the average results of the environmental stress-rupture durability tests. Complete tabulations of all the individual test data, including computed standard deviations, for both the static and environmental durability tests are presented in Appendix A. In addition to these tabulations, the environmental stress-rupture durability data are graphically illustrated in Figures 5 through 7.

1. STATIC LAP SHEAR TEST RESULTS

Examination of the lap shear test results in Tables 1, 3, and 5, obtained after no pre-test conditioning, indicate that for all test temperatures up to 140°F (60°C), the failures were 100% cohesive for all three batches of adhesive, and the strengths for all three batches were very consistent. Since all of these failures were cohesive, no comparison of the surface treatments (acid etch and anodized) is available from these particular data. At 250°F (121°C), however, the first batch of adhesive exhibited a considerably higher strength than either the second or third batches. Comparison of these data with vendor (Bloomingdale) data indicates that the 3770 psi* (26.0 MPa) value for the first batch appears to be the anomalous number. Since the first and second batches of adhesive produced comparable degrees of cohesive failure (85% and 75%, respectively) but substantially different strengths, batch-to-batch differences in the adhesive might be indicated.

The lap shear strengths obtained after 30 and 100-day humidity agings without load indicate a significant difference between 120°F (49°C) and 140°F (60°C) aging condition. The strength of the specimens aged at 120°F (49°C) was about the

*Vendor data indicates approximately 2000 psi (13.8 MPa) at 250°F (121°C).

TABLE 1

SINGLE LAP SHEAR STRENGTH OF ADHESIVE JOINTS

Adherends: 2024T3 bare aluminum
Surface Preparation: Optimized FPL etch
Surface Primer: BR 127
Adhesive: FM-123-2 (First batch)

Test Temperature (°F)	(°C)	Pre-Test Conditioning [days at 140°F (60°C) & 95% R.H.-No Load]	Ultimate Strength (psi)	(MPa)	Failure Mode (% Coh.)	Number of Specimens Represented
72	22	None	5850	40.3	100	6
140	60	None	5020	34.6	100	12
250	121	None	3770	26.0	85	6
72	22	30	4500	31.5	99	6
72	22	100	4170	29.1	80	6

TABLE 2

ENVIRONMENTAL STRESS-RUPTURE LAP SHEAR BEHAVIOR OF ADHESIVE JOINTS

Adherends: 2024T3 bare aluminum
Surface Preparation: Optimized FPL etch
Surface Primer: BR 127
Adhesive: FM-123-2 (First batch)
Exposure Environment: 140°F(60°C) and 95% R.H.

Joint Shear Stress During Exposure (psi)	(MPa)	(% of 140°F dry ultimate)	Time to Failure (hrs)	Residual Lap Shear Strength (psi)	(MPa)	Failure Mode (% Coh.)	Number of Specimens Represented
4020	27.7	80	0*	---	---	60	3
3510	24.2	70	17.9	---	---	80	4
3010	20.7	60	57.9	---	---	80	4
2510	17.3	50	99.7	---	---	75	4
2010	13.8	40	486.7	---	---	75	4
1510	10.4	30	658.1	---	---	30	5
1000	6.9	20	1893.0	---	---	30	3

*All specimens failed immediately upon or during load application.

TABLE 3

SINGLE LAP SHEAR STRENGTH OF ADHESIVE JOINTS

Adherends:	2024T3 bare aluminum
Surface Preparation:	Phosphoric Acid Anodized
Surface Primer:	BR 127
Adhesive:	FM-123-2 (Second batch)

Test Temperature (°F)	(°C)	Pre-Test Conditioning [days at 140°F (60°C) & 95% R.H.-No Load]	Ultimate Strength (psi)	(MPa)	Failure Mode (% Coh.)	Number of Specimens Represented
72	22	None	5770	39.8	100	6
120	49	None	5100	34.4	100	9
250	121	None	2500	17.2	75	6
72	22	30	5520	38.1	100	6
72	22	100	5530	38.1	99	6

TABLE 4

ENVIRONMENTAL STRESS-RUPTURE LAP SHEAR BEHAVIOR OF ADHESIVE JOINTS

Adherends:	2024T3 bare aluminum
Surface Preparation:	Phosphoric Acid Anodized
Surface Primer:	BR 127
Adhesive:	FM-123-2 (Second batch)
Exposure Environment:	120°F(49°C) and 95% R.H.

Joint Shear Stress During Exposure (psi)	(MPa)	(% of 120°F dry ultimate)	Time to Failure (hrs)	Residual Lap Shear Strength[2] (psi)	(MPa)	Failure Mode (% Coh.)	Number of Specimens Represented
4100	28.2	80	0.11	---	---	100	3
3590	24.7	70	4	---	---	100	3
3080	21.2	60	111	---	---	100	3
2570	17.7	50	812	---	---	100	5
2050	14.1	40	2147	5710[3]	39.4	100	3
1540	10.6	30	2400[1]	5090	35.1	100	3
1030	7.1	20	2400[1]	5040	34.1	100	3

[1]Joints did not fail within 2400-hour exposure period and were removed for residual strength testing.
[2]All residual strengths were obtained at 72°F (22°C).
[3]Two specimens survived 2400-hour exposure period.

TABLE 5

SINGLE LAP SHEAR STRENGTH OF ADHESIVE JOINTS

Adherends: 2024T3 bare aluminum
Surface Preparation: Optimized FPL etch
Surface Primer: BR 127
Adhesive: FM-123-2 (Third batch)

Test Temperature (°F)	(°C)	Pre-Test Conditioning [days at 140°F (60°C) & 95% R.H.-No Load]	Ultimate Strength (psi)	(MPa)	Failure Mode (% Coh.)	Number of Specimens Represented
72	22	None	5920	40.8	100	5
120	49	None	5390	37.2	100	10
250	121	None	2220	15.3	25	5
72	22	30	5440	37.5	85	5
72	22	100	5730	39.5	95	5

TABLE 6

ENVIRONMENTAL STRESS-RUPTURE LAP SHEAR BEHAVIOR OF ADHESIVE JOINTS

Adherends: 2024T3 bare aluminum
Surface Preparation: Optimized FPL etch
Surface Primer: BR 127
Adhesive: FM-123-2 (Third batch)
Exposure Environment: 120°F(49°C) and 95% R.H.

Joint Shear Stress During Exposure (psi)	(MPa)	(% of 120°F dry ultimate)	Time to Failure (hrs)	Residual Lap Shear Strength[2] (psi)	(MPa)	Failure Mode (% Coh.)	Number of Specimens Represented
3230	22.3	60	1939	---	---	75	3
2160	14.9	40	2400[1]	5790	39.9	98	3
1620	11.2	30	2400[1]	5690	39.2	100	2
1080	7.4	20	2400[1]	5720	39.4	100	3

[1]Joints did not fail within 2400-hour exposure period and were removed for residual strength testing.
[2]All residual strengths were obtained at 72°F (22°C).

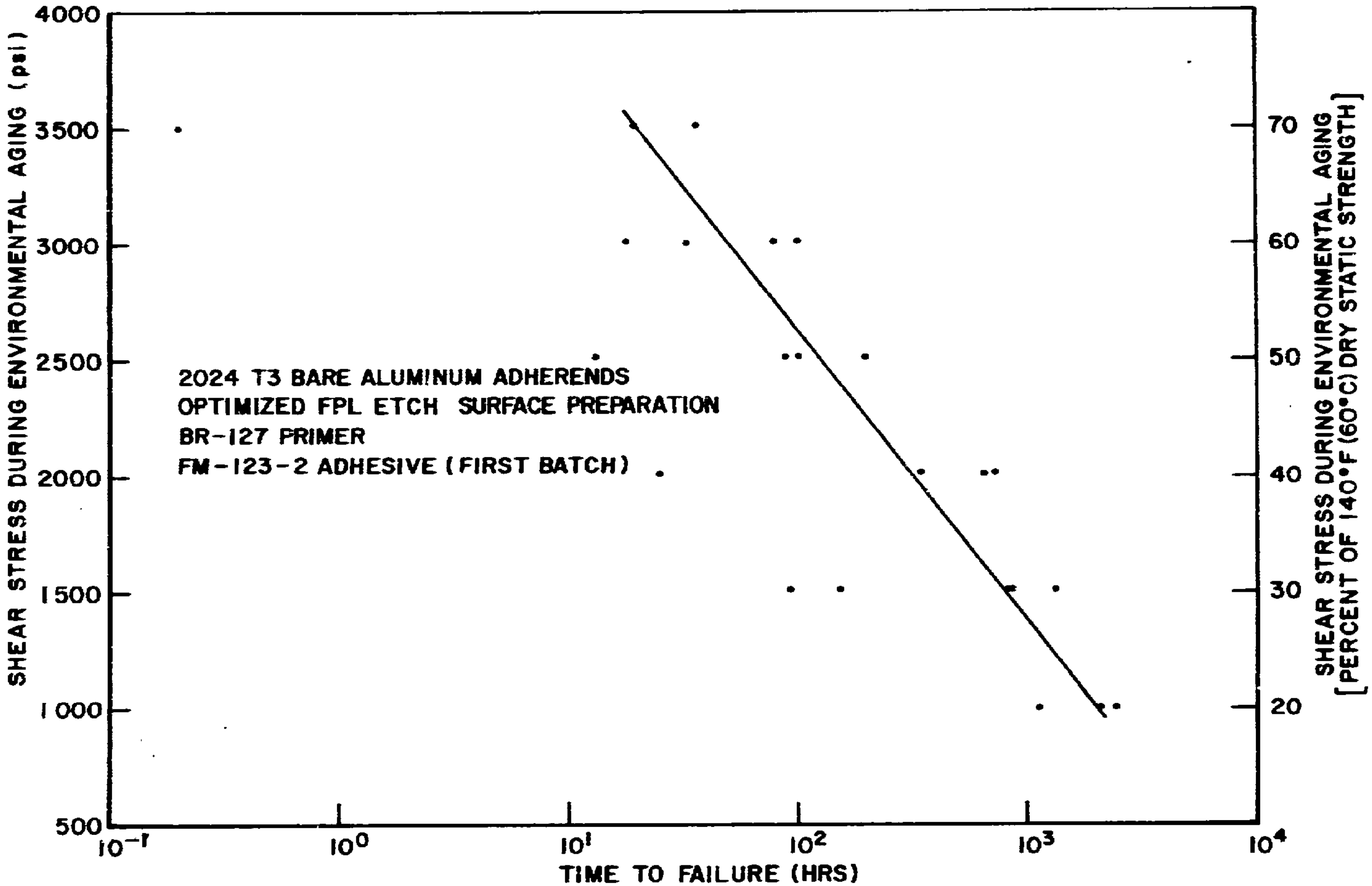


Figure 5. Environmental Stress-Rupture Time-to-Failure Behavior of Single Lap Shear Adhesive Joints at 140°F (60°C) and 95-100% R.H.

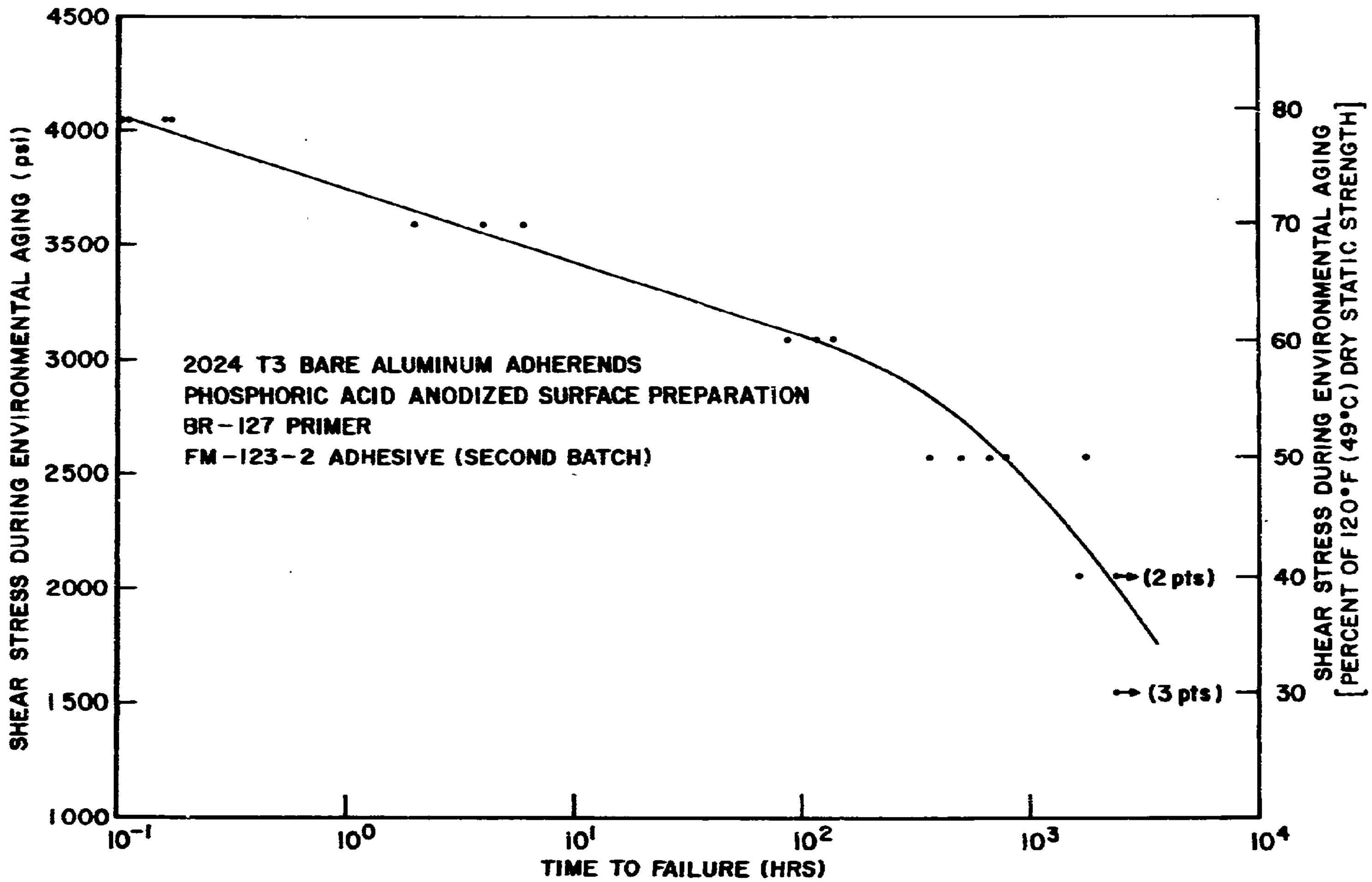


Figure 6. Environmental Stress-Rupture Time-to-Failure Behavior of Single Lap Shear Adhesive Joints at 120°F (49°C) and 95-100% R.H.

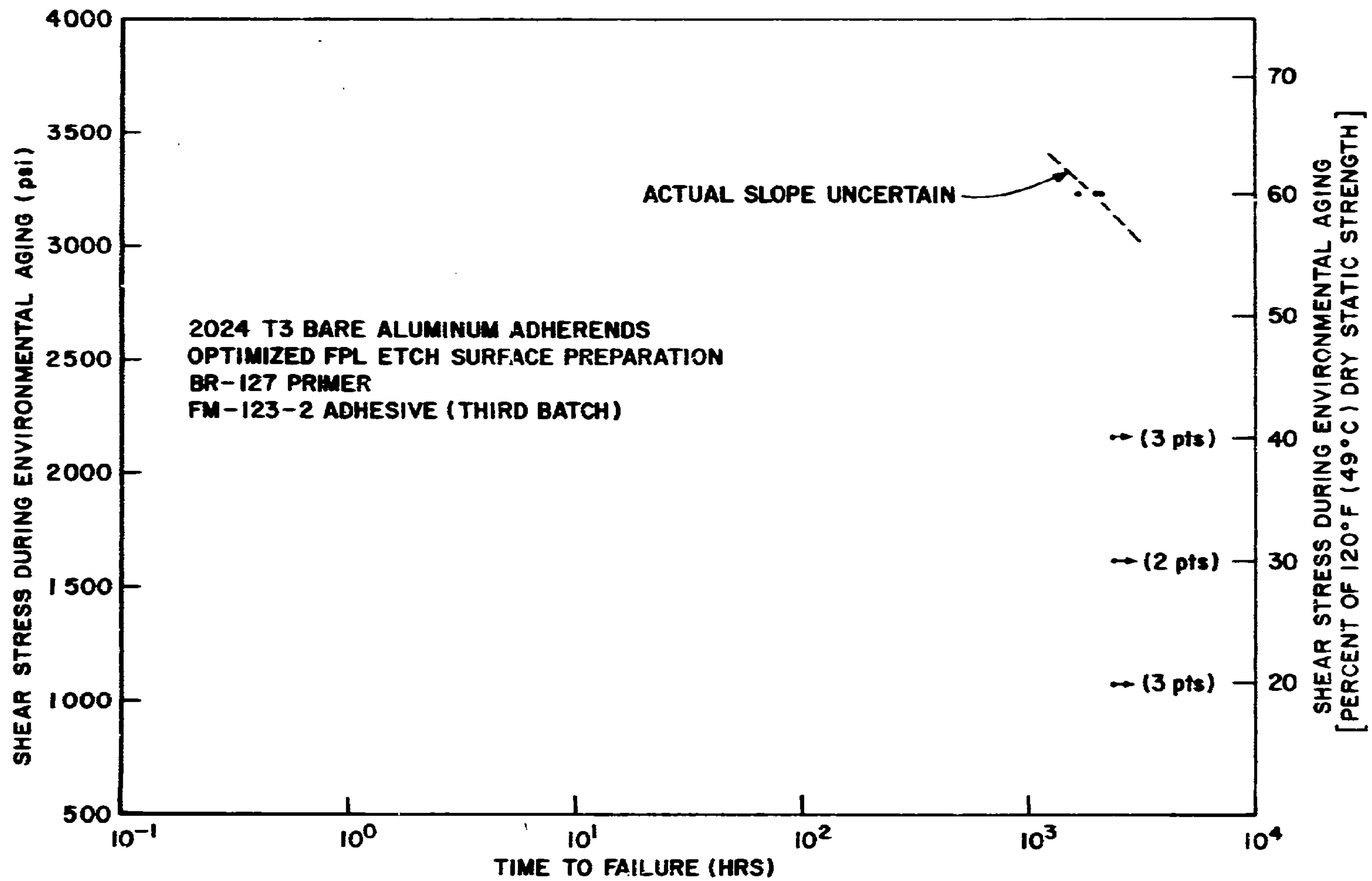


Figure 7. Environmental Stress-Rupture Time-to-Failure Behavior of Single Lap Shear Adhesive Joints at 120°F (49°C) and 95-100% R.H.

same as the unaged 72°F (22°C) strength while specimens aged at 140°F (60°C) lost nearly 25% of their 72°F (22°C) strength. The anodized surface condition produced almost completely cohesive failures for these tests while the acid etched surface condition produced predominately cohesive failures but still with significantly more adhesive interfacial failure than the anodized specimens.

2. ENVIRONMENTAL STRESS-RUPTURE TEST RESULTS

At first glance, these results appear difficult to interpret. The results for the second and third adhesive batches, obtained in a 120°F (49°C) environment, exhibit 100% cohesive failure on the specimens with an anodized surface treatment and predominately, but not complete cohesive failure on the specimens with an acid etched surface treatment. In spite of the greater overall cohesive failure behavior, the specimens on the anodized surfaces (second batch of adhesive) exhibited considerably lower levels of durability during the environmental stress-rupture exposures than the specimens on the acid etched surfaces (third batch of adhesive). This is clearly illustrated by the comparative data presented in Figure 8. Inasmuch as the failure modes of these specimens indicate that the failures were within the adhesive itself rather than along the bonded adhesive/substrate interface, little can be concluded from these data regarding the comparative merits of anodization vs. acid etching as a surface preparation procedure. The difference in durability between the two sets of specimens, both of which exhibited predominately or complete cohesive failure, would appear then to be ascribable to batch-to-batch variations in the adhesive itself. A second possibility, one which was discussed in AFML-TR-78-35, Part I and was first described by Marceau*, may also be clouding the data.

*J.A. Marceau, "An SEM Analysis of Adhesive Primer Oriented Bond Failures on Anodized Aluminum," presented at 23rd National SAMPE Symposium, Anaheim, Calif., May 2-4, 1978.

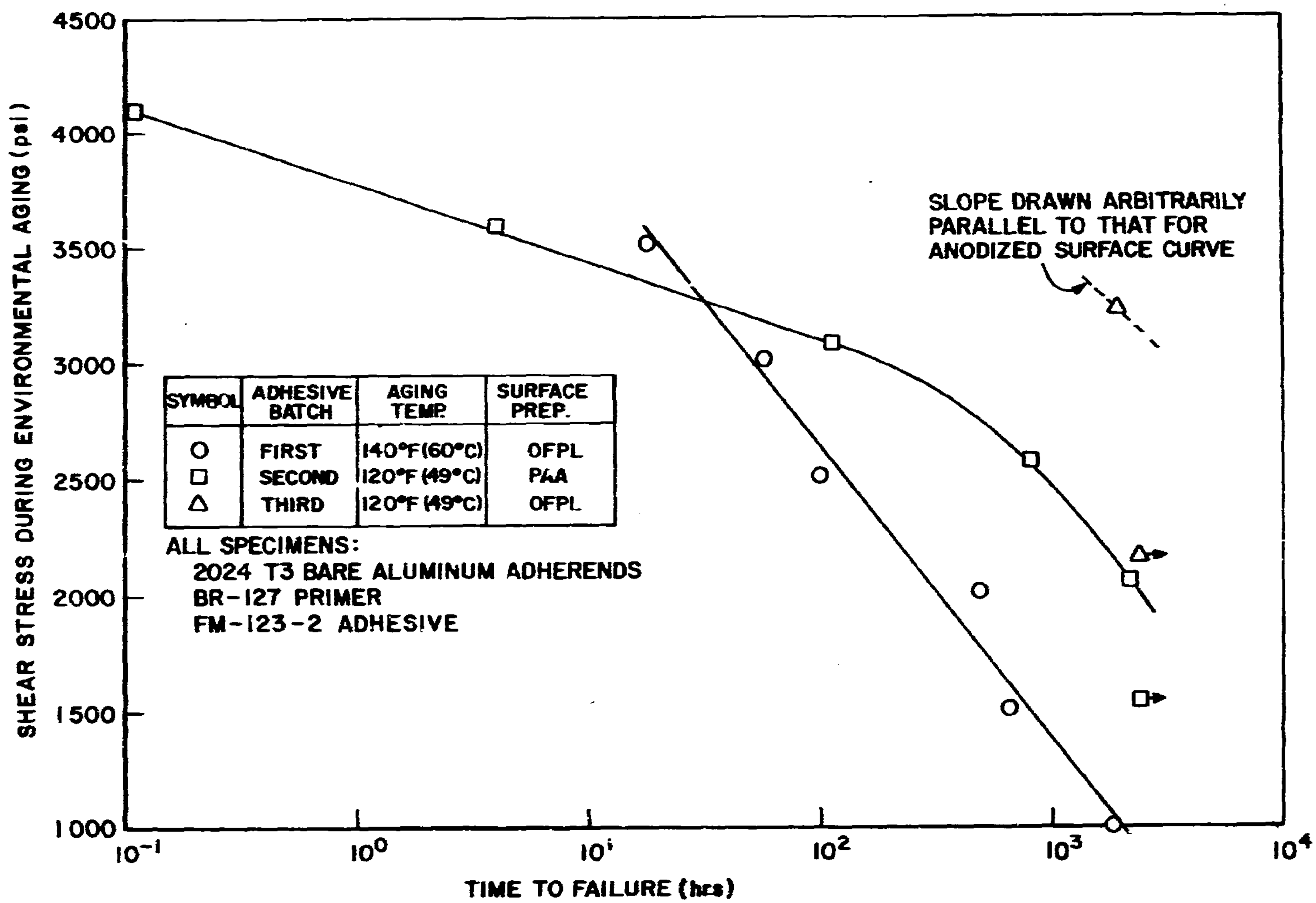


Figure 3. Comparative Environmental Stress-Rupture Behavior of Single Lap Shear Adhesive Joints

Marceau hypothesized that when the aluminum is sufficiently strained during specimen loading, the surface oxide layer, being of a much higher modulus and lower ultimate strain capability, will fracture. For the types and sizes of specimens employed here, this oxide fracture would be expected to occur whenever the lap shear stress reached the general vicinity of about 3000 psi (20.65 MPa). It can be readily observed that the time-to-failure curve for the anodized surface in Figure 8 exhibits a rather distinct change of slope in the 3000 psi (20.65 MPa) region, implying that perhaps the degradation mechanism which leads to failure is different at stresses below this level than it is at higher stresses. This still leaves unsettled, however, the question of why, at 120°F (49°C), the anodized specimens exhibit shorter lifetimes than the OFPL etched specimens in the 2000-2500 psi (13.78-17.2 MPa) region. This may lead back to the batch-to-batch variation question.

A question which still remains is, why do the OFPL etched specimens tested for durability at 140°F (60°C) exhibit longer lifetimes than the PAA specimens tested at 120°F (49°C) at stresses above 3500 psi (24.1 MPa)? Perhaps the substantially higher 250°F (121°C) dry static strength of the first batch of adhesive indicates that this batch has an inherently greater resistance to degradation than the second. Perhaps the considerably thicker and more porous oxide layer on the anodized specimens leads to fracture of this layer, and a concomitant elbow in the time-to-failure curve, at a lower stress level than on the acid etched specimens. Neither of these possibilities, or any other for that matter, can be advanced as an unequivocal explanation for this behavior. At any rate, at the lower stress levels, below which neither oxide would be expected to fracture, the effect of a higher temperature aging condition is clearly evident. Considerably more interfacial adhesive failures were observed in the 140°F (60°C) durability exposures than in the 120°F (49°C) exposures. The progressively increasing degree of adhesive failure (decreasing percent cohesive failure) at the longer lifetimes indicate that when under stress at 140°F (60°C)

and 95-100% R.H. the OFPL etch surface is being slowly degraded and that, if the adhesive does not fail cohesively, the interface will eventually deteriorate to the point where it is weaker than the adhesive layer and will cause joint failure. Since the 120°F (49°C) durability agings on the OFPL etched surfaces failed completely cohesively at the lower stress-longer time conditions, the marked effect of temperature upon the degradation rate of the adherend adhesive interface is clearly evident.

SECTION V

CONCLUSIONS

1. The environmental stress-rupture durability testing conducted in this program indicate that at single lap shear stress levels below 2000 psi (13.9 MPa), FM-123-2 adhesive is capable of providing very long service lives (>2000 hrs) at 120°F (49°C) and 95-100% R.H. This stress level corresponds to about 40% of the ultimate strength of this adhesive in 120°F (49°C), dry static tests.

2. FM-123-2 adhesive, when used in conjunction with BR127 primer, produced high levels of bonding and long lifetimes on both PAA and OFPL etched surfaces when the stress level was below that at which the adherend oxide layer fractures.

3. It appears as if the environmental stress-rupture degradation mechanism leading to failure of the specimens prepared with phosphoric acid anodized surfaces changes in the general region of about 3000 psi (20.65 MPa). This is about where one would expect the surface oxide layer to fracture on this type of specimen. Data was insufficient to detect such a phenomena on the optimized FPL etched specimens.

4. The effect of a higher aging temperature upon the stress-durability of an adhesive bond prepared with an optimized FPL etched surface, BR127 primer, and FM-123-2 adhesive appears to be more detrimental to the interface than to the adhesive itself.

5. Some anomalies in the data indicate that batch-to-batch variations in the adhesive material may have been encountered. It would be advisable to prepare all specimens from the same batch of material. We were unable to do this since the program reported here was actually conducted in several segments over a period of two years.

APPENDIX A
COMPLETE TEST DATA

TABLE A.1

LAP SHEAR STRENGTH OF ADHESIVE JOINTS

Adherends:	**0.250 inch (0.64 cm) thick 2024T3 bare aluminum**
Surface Preparation:	**Optimized FPL etch**
Surface Primer:	**BR-127**
Adhesive:	**FM-123-2 (First batch of material received - April, 1976)**

Test Temperature (°F)	(°C)	Pre-Test Conditioning [days @ 140°F (60°C) and 95% R.H. No Load]	Ultimate Strength (psi)	(MPa)	Failure Mode (% Coh. Failure)
72	22	None	5990	41.3	100
72	22	None	5700	39.3	100
72	22	None	6100	42.1	100
72	22	None	5920	40.8	100
72	22	None	5770	39.8	100
72	22	None	5600	38.6	100
Average			5850	40.3	100
Std. Dev.			190	1.3	0
140	60	None	5120	35.3	100
140	60	None	4760	32.8	100
140	60	None	4890	33.7	100
140	60	None	4960	34.2	100
140	60	None	5110	35.2	100
140	60	None	4890	33.7	100
140	60	None	5020	34.6	100
140	60	None	4770	32.9	100
140	60	None	5460	37.7	100
140	60	None	5280	36.4	100
140	60	None	5300	36.5	100
140	60	None	4630	31.9	100
Average			5020	34.6	100
Std. Dev.			250	1.7	0
250	121	None	3560	24.5	60
250	121	None	3740	25.8	95
250	121	None	3820	26.3	80
250	121	None	4030	27.8	80
250	121	None	3450	23.8	100
250	121	None	4010	27.6	100
Average			3770	26.0	85
Std. Dev.			235	1.6	15
72	22	30	4990	34.9	100
72	22	30	4420	30.8	100
72	22	30	4980	34.8	100
72	22	30	3220	22.5	100
72	22	30	4570	31.9	100
72	22	30	4830	33.8	95
Average			4500	31.5	99
Std. Dev.			670	4.6	2
72	22	100	4740	33.1	100
72	22	100	4640	32.4	100
72	22	100	4670	32.6	90
72	22	100	2910	20.3	50
72	22	100	4370	30.5	100
72	22	100	3710	25.9	40
Average			4170	29.1	80
Std. Dev.			725	5.0	27

TABLE A.2

LAP SHEAR STRENGTH OF ADHESIVE JOINTS

Adherends: 0.250 inch (0.64 cm) thick 2024T3 bare aluminum
Surface Preparation: Phosphoric Acid Anodized
Surface Primer: BR-127
Adhesive: FM-123-2 (Second batch of material received - Sept., 1976)

Test Temperature (°F)	Test Temperature (°C)	Pre-Test Conditioning [days @ 120°F (49°C) and 95% R.H. No Load]	Ultimate Strength (psi)	Ultimate Strength (MPa)	Failure Mode (% Coh. Failure)
72	22	None	5620	38.7	100
72	22	None	5760	39.7	100
72	22	None	5820	40.1	100
72	22	None	5670	39.1	100
72	22	None	5890	40.6	100
72	22	None	5870	40.5	100
Average			5770	39.8	100
Std. Dev.			100	0.8	0
120	49	None	5390	37.2	100
120	49	None	4800	33.1	100
120	49	None	4980	34.3	100
120	49	None	3910	27.0	100
120	49	None	5270	36.3	100
120	49	None	5210	35.9	100
120	49	None	5920	33.9	100
120	49	None	5070	35.0	100
120	49	None	5380	37.1	100
Average			5100	34.4	100
Std. Dev.			550	3.1	0
250	121	None	2930	20.2	100
250	121	None	3360	23.2	90
250	121	None	1470	10.1	25
250	121	None	2250	15.5	80
250	121	None	2530	17.4	80
250	121	None	2430	16.8	80
Average			2500	17.2	75
Std. Dev.			640	4.4	25
72	22	30	5460	37.6	100
72	22	30	5380	37.1	100
72	22	30	5580	38.5	100
72	22	30	5760	39.7	100
72	22	30	5480	37.8	100
72	22	30	5480	37.8	100
Average			5520	38.1	100
Std. Dev.			130	0.9	0
72	22	100	5800	39.9	100
72	22	100	5320	36.7	100
72	22	100	5620	38.7	100
72	22	100	5610	38.7	100
72	22	100	5370	37.0	95
72	22	100	5470	37.7	100
Average			5530	38.1	99
Std. Dev.			180	1.2	2

TABLE A.3

LAP SHEAR STRENGTH OF ADHESIVE JOINTS

Adherends: 0.250 inch (0.64 cm) thick 2024T3 bare aluminum
Surface Preparation: Optimized FPL etch
Surface Primer: BR-127
Adhesive: FM-123-2 (Third batch of material received - Nov., 1977)

Test Temperature (°F)	(°C)	Pre-Test Conditioning [days @ 120°F(49°C) and 95% R.H. No Load]	Ultimate Strength (psi)	(MPa)	Failure Mode (% Coh. Failure)
72	22	None	5950	41.0	100
72	22	None	5710	39.3	100
72	22	None	6000	41.3	100
72	22	None	5990	41.3	100
72	22	None	5970	41.1	100
Average			5920	40.8	100
Std. Dev.			120	0.8	0
120	49	None	5160	35.6	100
120	49	None	5290	33.9	100
120	49	None	5530	38.1	100
120	49	None	5290	33.9	100
120	49	None	5610	38.7	100
120	49	None	5720	39.4	100
120	49	None	5380	37.1	100
120	49	None	5610	38.7	100
120	49	None	5140	35.4	100
120	49	None	5130	35.3	100
Average			5390	37.2	100
Std. Dev.			220	1.5	0
250	121	None	2390	16.5	25
250	121	None	2110	14.5	25
250	121	None	2300	15.8	25
250	121	None	2440	16.8	25
250	121	None	1840	12.7	25
Average			2220	15.3	25
Std. Dev.			245	1.7	0
72	22	30	5420	37.3	90
72	22	30	4630	31.9	60
72	22	30	5650	39.0	90
72	22	30	5820	40.1	100
72	22	30	5700	39.3	90
Average			5440	37.5	85
Std. Dev.			480	3.3	15
72	22	100	5970	41.2	100
72	22	100	5710	39.3	75
72	22	100	5730	39.5	100
72	22	100	5570	38.4	100
72	22	100	5850	40.3	100
Average			5730	39.5	95
Std. Dev.			100	0.8	10

TABLE A.4

ENVIRONMENTAL STRESS-RUPTURE LAP SHEAR BEHAVIOR OF ADHESIVE JOINTS

Adherends: 0.250 inch (0.64 cm) thick 2024T3 bare aluminum
Surface Preparation: Optimized FPL etch
Surface Primer: BR-127
Adhesive: FM-123-2 (First batch of material received - April, 1976)
Exposure Environment: 140°F(60°C) and 95% R.H.

Joint Shear Stress During Exposure (psi)	(MPa)	(% of 140°F dry ultimate)	Time to Failure (hrs)	Residual Lap Shear Strength (psi)	(MPa)	Failure Mode (% Cohesive)
4020	27.7	80	0*	---	---	60
4020	27.7	80	0*	---	---	60
4020	27.7	80	0*	---	---	60
Average			0	---	---	60
Std. Dev.			---	---	---	0
3510	24.2	70	36.3	---	---	95
3510	24.2	70	19.6	---	---	100
3510	24.2	70	0.2	---	---	60
3510	24.2	70	15.6	---	---	60
Average			17.9	---	---	80
Std. Dev.			14.8	---	---	20
3010	20.7	60	18.0	---	---	100
3010	20.7	60	79.5	---	---	90
3010	20.7	60	101.0	---	---	90
3010	20.7	60	33.0	---	---	50
Average			57.9	---	---	80
Std. Dev.			38.9	---	---	20
2510	17.3	50	101.4	---	---	100
2510	17.3	50	195.4	---	---	90
2510	17.3	50	13.1	---	---	50
2510	17.3	50	88.9	---	---	50
Average			99.7	---	---	75
Std. Dev.			74.8	---	---	25
2010	13.8	40	342.2	---	---	60
2010	13.8	40	25.1	---	---	40
2010	13.8	40	652.3	---	---	100
2010	13.8	40	727.1	---	---	100
Average			486.7	---	---	75
Std. Dev.			389.6	---	---	30
1510	10.4	30	823.7	---	---	80
1510	10.4	30	152.7	---	---	20
1510	10.4	30	1359.3	---	---	40
1510	10.4	30	861.9	---	---	0
1510	10.4	30	93.0	---	---	10
Average			658.1	---	---	30
Std. Dev.			532.8	---	---	30
1000	6.9	20	2449.0	---	---	40
1000	6.9	20	2106.3	---	---	10
1000	6.9	20	1125.0	---	---	40
Average			1893.0	---	---	30
Std. Dev.			687.0	---	---	15

*Specimen failed immediately upon or during load application.

TABLE A.5

ENVIRONMENTAL STRESS-RUPTURE LAP SHEAR BEHAVIOR OF ADHESIVE JOINTS

Adherends:	0.250 inch (0.64 cm) thick 2024T3 bare aluminum
Surface Preparation:	Phosphoric Acid Anodized
Surface Primer:	BR-127
Adhesive:	FM-123-2 (Second batch of material received - Sept., 1976)
Exposure Environment:	120°F(49°C) and 95% R.H.

Joint Shear Stress During Exposure (psi)	(MPa)	(% of 120°F dry ultimate)	Time to Failure (hrs)	Residual Lap Shear Strength (psi)	(MPa)	Failure Mode (% Cohesive)
4100	28.2	80	0.17	---	---	100
4100	28.2	80	0.00	---	---	100
4100	28.2	80	0.17	---	---	100
Average			0.11	---	---	100
Std. Dev.			0.10	---	---	0
3590	24.7	70	6	---	---	100
3590	24.7	70	2	---	---	100
3590	24.7	70	4	---	---	100
Average			4	---	---	100
Std. Dev.			2	---	---	0
3080	21.2	60	118	---	---	100
3080	21.2	60	138	---	---	100
3080	21.2	60	77	---	---	100
Average			111	---	---	100
Std. Dev.			31	---	---	0
2570	17.7	50	788	---	---	100
2570	17.7	50	362	---	---	100
2570	17.7	50	678	---	---	100
2570	17.7	50	1736	---	---	100
2570	17.7	50	495	---	---	100
Average			812	---	---	100
Std. Dev.			542	---	---	0
2050	14.1	40	2400	5630	38.8	100
2050	14.1	40	2400	5780	39.9	100
2050	14.1	40	1640	---	---	100
Average			2147	5710	39.4	100
Std. Dev.			439	---	---	0
1540	10.6	30	2400	5160	35.6	100
1540	10.6	30	2400	4950	34.1	100
1540	10.6	30	2400	5160	35.6	100
Average			2400	5090	35.1	100
Std. Dev.			0	---	---	0
1030	7.1	20	2400	5160	35.6	100
1030	7.1	20	2400	5080	35.0	100
1030	7.1	20	2400	4880	33.6	100
Average			2400	5040	34.8	100
Std. Dev.			0	---	---	0

TABLE A.6

ENVIRONMENTAL STRESS-RUPTURE LAP SHEAR BEHAVIOR OF ADHESIVE JOINTS

Adherends:	0.250 inch (0.64 cm) thick 2024T3 bare aluminum
Surface Preparation:	Optimized FPL etch
Surface Primer:	BR-127
Adhesive:	FM-123-2 (Third batch of material received - Nov., 1977)
Exposure Environment:	120°F(49°C) and 95% R.H.

Joint Shear Stress During Exposure (psi)	(MPa)	(% of 120°F dry ultimate)	Time to Failure (hrs)	Residual Lap Shear Strength (psi)	(MPa)	Failure Mode (% Cohesive)
3230	22.3	60	1672	---	---	50
3230	22.3	60	2089	---	---	75
3230	22.3	60	2057	---	---	100
Average			1939	---	---	75
Std. Dev.			232	---	---	25
2160	14.9	40	2400	5840	40.2	100
2160	14.9	40	2400	5900	40.7	95
2160	14.9	40	2400	5640	38.9	100
Average			2400	5790	39.9	98
Std. Dev.			0	140	1.0	3
1620	11.2	30	2400	5350	36.9	100
1620	11.2	30	2400	6020	41.5	100
Average			2400	5690	39.2	100
Std. Dev.			---	---	---	---
1080	7.4	20	2400	5820	40.1	100
1080	7.4	20	2400	5960	41.1	100
1080	7.4	20	2400	5380	37.1	100
Average			2400	5720	39.4	100
Std. Dev.			0	310	2.1	0

APPENDIX B

SURFACE PREPARATION PROCEDURES

APPENDIX B

SURFACE PREPARATION PROCEDURES

The specimens prepared and tested in this investigation were made with either an optimized FPL etch or with a phosphoric acid anodized surface preparation. Each of these are described below. The referenced BAC numbers refer to processing specifications developed by The Boeing Company.

1. OPTIMIZED FPL ETCH

The stepwise procedure used for this surface is:

(1) Scrub adherend surface with a nonchlorinated detergent in tap water, rinse, and dry.

(2) Wipe adherend surface with MEK and dry.

(3) Vapor degrease in trichloroethylene according to BAC 5408.

(4) Acid etch with the solutions and procedures contained in BAC 5514 for optimized FPL etch.

(5) Rinse immediately in continuously flowing tap water for ten minutes and dry with an air heat gun.

(6) Apply primer within 1/2 hour.

2. PHOSPHORIC ACID ANODIZATION

The stepwise procedure for this surface is:

(1) Scrub adherend surface with a nonchlorinated detergent in tap water, rinse, and dry.

(2) Wipe adherend surface with MEK and dry.

(3) Vapor degrease in trichloroethylene according to BAC 5408.

(4) Immerse in a deoxidizing alkaline wash of Oakite #164 at 140°F (60°C) for ten minutes.

(5) Rinse with continuously flowing tap water for ten minutes.

(6) Acid etch with an Oakite #34/sulfuric acid solution for one to three minutes at 72°F (22°C).

(7) Rinse with continuously flowing tap water for ten minutes.

(8) Phosphoric acid anodize the adherends for 25 minutes at 10 $\pm$ 1 volts.

(9) Rinse with continuously flowing tap water for ten minutes and dry panels with an air heat gun. The air flow from this heat gun is at a temperature of 200-250°F (93-121°C) although the part never reaches that temperature since the air flow is moved back and forth over the surface for only about one minute.

(10) Apply primer within 1/2 hour.

APPENDIX C

PANEL BONDING PROCEDURES

APPENDIX C

PANEL BONDING PROCEDURES

The stepwise procedure used to cure the panels from which individual specimens were subsequently machined is:

(1) Layup primed panels and adhesive film into assembly required for final specimens.

(2) Place layup assembly in autoclave at room temperature.

(3) Pull a vacuum on the bagged assembly.

(4) Apply 45 $\pm$ 5 psi (310 $\pm$ 34 KPa) over the bladder and then release the vacuum.

(5) Heat the autoclave at 5-7°F/min to 350°F (177°C).

(6) Hold at 350°F (177°C) for 60 minutes.

(7) Cool the autoclave to below 200°F (93°C), maintaining the 45 $\pm$ 5 psi (310 $\pm$ 34 KPa) over the bladder.

(8) Release pressure and remove the panel from the autoclave.

APPENDIX D

SPECIMEN PREPARATION PROCEDURES

APPENDIX D

SPECIMEN PREPARATION PROCEDURES

The as-fabricated panels are 16 inches (40.6 cm) wide when bonded and are first cut into 13 individual specimens on a bandsaw. These rough-cut specimens are then finish milled down to their final 1 inch (2.54 cm) wide by 7 inches (17.8 cm) long dimension. Holes are drilled through the ends for mounting in the test grips as well as for specimen location in a machining fixture when the specimens are slotted across their width. The slots are cut across the specimens to provide the lap joint. These slots are machined down to, but not through, the adhesive layer. Finally, the ends of the specimen are machined down to a 0.250 inch (0.64 cm) thickness to fit into the test grips on the environmental stress-rupture durability tester.

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